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NIST/NRC-Canada Interlaboratory Comparison of Guarded Hot Plate Measurements: 1993-1997

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Abstract

Thermal conductivity measurements from an international interlaboratory study between the United States and Canada are presented for two materials, glass-fiber board and fibrous alumina silica. The measurements were determined using guarded hot plate apparatus at the National Institute of Standards and Technology and at the National Research Council Canada. The nominal bulk densities and thicknesses for the specimens of glass-fiber board and fibrous alumina silica were 160 kg/m^3 and 25.1 mm, and 290 kg/m^3 and 25.2 mm, respectively. Measurements of thermal conductivity were conducted over a temperature range of 273 K to 340 K. Statistical regression analyses of the data are presented.

Keywords: alumina silica, building technology, glass fiber, guarded hot plate apparatus, insulation, interlaboratory, international, regression, statistics, steady state, thermal conductivity, thermal resistance, uncertainty

Introduction

In North America, manufacturers of thermal insulation must satisfy the thermal performance requirements promulgated by the governments of the United States and Canada in order to market their products in both countries. Consequently, these industries depend heavily on national standards for thermal resistance issued by the United States and by Canada. In the United States, the responsibility for national standards has traditionally been maintained by the National Institute of Standards and Technology (NIST) and in Canada, by the National Research Council Canada (NRC-Canada). Recently, a question concerning the agreement of thermal resistance measurements between NIST and NRC-Canada has arisen. To address this issue, NIST and NRC-Canada have completed an interlaboratory study between their respective guarded hot plate apparatus. This study is intended as a first step (or pilot run) for future comparisons between the laboratories.

The guarded hot plate apparatus is considered an absolute method for the determination of thermal transmission properties of heat insulators under steady-state conditions. The test method has been standardized for several years; see, for example, ASTM Test Method C 177 [1] or ISO 8302 [2]. Both test methods allow some latitude in the design of the apparatus. An assessment of apparatus errors by theoretical treatment, i.e., error propagation formula, can yield a relative combined standard uncertainty ($k = 1$) of approximately ± 0.5 % to ± 1.0 % for a properly designed apparatus. The stated imprecision for standard test methods is higher, however, on the order of ± 2 % to ± 5 % [1,2] at ambient conditions for homogeneous materials. These imprecision values for standard test methods have been derived from empirical test data obtained from different laboratories and include the effects of other factors such as operator, specimen homogeneity, or equipment variations.

The interlaboratory study between NIST and NRC-Canada was initiated in 1993, completed in 1997, and included two materials, glass-fiber board and fibrous alumina silica. The first material was selected from Standard Reference Material (SRM) 1450c, Fibrous Glass Board [3]. The second material was originally part of an ASTM C-16.30 Interlaboratory Study of high-temperature guarded hot plate apparatus. The data presented herein for fibrous alumina silica are used with permission of the ASTM C-16.30 Task Group. A single pair of specimens of each material was circulated between both laboratories; therefore, this comparison represents a round-robin in the true sense. Values of apparent[1] thermal conductivity were reported over a temperature range of 273 K to 340 K. This report describes the test materials, test method and equipment, experimental results, and statistical analyses of the thermal conductivity data.

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[1]The thermal transmission properties of heat insulators determined from standard test methods typically include several mechanisms of heat transfer, including conduction, radiation, and possibly convection. For that reason, some experimentalists will include the adjective "apparent" when describing thermal conductivity of thermal insulation. However, for brevity, the term thermal conductivity will be used in this report.

Experimental

Materials

In this study, a single pair of glass-fiber board and fibrous alumina silica specimens were circulated to both laboratories. The pair of glass-fiber board specimens were selected from the material lot used to develop SRM 1450c [3] and the pair of fibrous alumina silica specimens were provided as part of an ASTM C-16.30 Interlaboratory Study. Table 1 summarizes the average bulk density (ρ), measured in air, and specimen thickness (L) of the specimens. The small difference in bulk density for glass-fiber board was due to local inhomogeneities in the specimens [3]. Because the apparatus at NIST and NRC-Canada required different size specimens, the specimens were tested first at NIST and subsequently cut to the necessary dimensions for the apparatus at NRC-Canada.

Table 1 -- Specimen Physical Properties

Laboratory	Glass-fiber board		Fibrous Alumina Silica	
	ρ	L	ρ	L
	($kg{\cdot}m^{-3}$)	(mm)	($kg{\cdot}m^{-3}$)	(mm)
NIST	158	25.11	288	25.19
NRC-Canada	160	25.08	288	25.21

Test Method and Apparatus

Measurements of thermal conductivity were determined in accordance with standard test methods, ASTM C 177 and/or ISO 8302 [1,2] which are summarized briefly here. Two specimens having nearly the same density, size, and thickness are placed on the two sides of the guarded hot plate and clamped securely by the cold plates. Ideally, the guarded hot plate and the cold plates provide constant temperature boundary conditions to the surfaces of the specimens. With proper guarding in the lateral direction, the apparatus is designed to provide one-dimensional heat flow (Q) through the meter area of the pair of specimens. Additional guarding is provided by a temperature controlled environmental chamber which ordinarily maintains the ambient air temperature at the same value as the mean temperature (T) of the hot and the cold plates.

Under steady-state conditions, measurements of thermal conductivity (λ) for the pair of specimens were determined using the following equation:

$$Q = \lambda 2A \frac{\Delta T}{L}, \qquad (1)$$

where Q is the heat flow through the meter area of the specimens [W]; $2A$ is the meter area normal to direction of heat flow, both sides [m^2]; ΔT is the temperature difference across specimens [K]; and, L is the in-situ thickness of the pair of specimens [m]. Values of λ were reported at the mean temperature (T) of the hot and cold plates, $T = \frac{1}{2}(T_h + T_c)$.

Table 2 summarizes the criteria of the guarded hot plate apparatus used in this study. The essential differences between the NIST and NRC-Canada apparatus were the plate sizes, plate geometry, and the techniques used to heat the guarded hot plate and measure the corresponding temperature.

Table 2 -- Guarded Hot Plate Apparatus			
Parameter	**NIST**	**NRC-Canada #1**	**NRC-Canada #2**
Test Standard	ASTM C 177, C 1043	ISO 8302, ASTM C 177	ASTM C 177
Plate Size	1016 mm diameter	610 mm square	610 mm square
Meter Plate Size	406 mm diameter	250 mm square	300 mm square
Plate Material	aluminum	aluminum	aluminum
Plate Emittance	0.89 (normal)	0.89	0.89
Type of heater	line-heat source [4]	distributed	distributed
Temperature	platinum RTD	thermocouple, Type T	thermocouple, Type T
Year of Service	1981	1990 to 1992	1953

Results

Thermal conductivity data for glass-fiber board are summarized in Table 3 and plotted as a function of mean temperature in Figure 1a. Each datum in Figure 1a includes uncertainty bars that represent

Table 3 -- Thermal Conductivity Measurements for Glass-fiber Board				
		T	**T**	**λ**
Laboratory	**Test**	**(°C)**	**(K)**	**($W \cdot m^{-1} \cdot K^{-1}$)**
NIST	1	7.0	280	0.0314
NIST	2	22.0	295	0.0331
NIST	4	37.0	310	0.0348
NIST	4	52.0	325	0.0366
NIST	5	67.0	340	0.0382
NRC-Canada #1	1	15.0	288	0.0329
NRC-Canada #1	2	24.0	293	0.0337
NRC-Canada #1	3	23.9	297	0.0339
NRC-Canada #1	4	24.0	297	0.0337
NRC-Canada #1	5	27.0	300	0.0340
NRC-Canada #1	6	30.7	304	0.0346
NRC-Canada #1	7	36.2	309	0.0352

the relative (expanded, $k = 2$) uncertainty estimate for the measurement, approximately ± 1.0 % for both laboratories. A small difference, on the order of 1 %, was observed for the two sets of data. Part of the difference (no more than 0.3 %) could be attributed to the differences in bulk density for the different specimen sizes (Table 1). Note, however, that the difference between the two sets of data was within the measurement uncertainties represented by the uncertainty bars. The line in Figure 1a represents a linear regression fit of the data treated collectively, as described later.

Thermal conductivity data for fibrous alumina silica are summarized in Table 4 and plotted as a function of mean temperature in Figure 1b. Again, each datum in Figure 1b includes uncertainty bars that represent a relative (expanded, $k = 2$) uncertainty estimate for the thermal conductivity measurement of both laboratories, approximately ± 1.0 %. In this case, the two sets of data essentially agree over the entire temperature range. The line in Figure 1b represents a linear regression fit of the data treated collectively, described later.

Table 4 -- Thermal Conductivity Measurements of Fibrous Alumina Silica

		T	T	λ
Laboratory	**Test**	**(°C)**	**(K)**	**($W \cdot m^{-1} \cdot K^{-1}$)**
NIST	1	5.0	278	0.0429
NIST	2	20.0	293	0.0449
NIST	3	25.0	298	0.0456
NIST	4	35.0	308	0.0468
NIST	5	50.0	323	0.0488
NRC-Canada #2	1	0.1	273	0.0426
NRC-Canada #2	2	12.1	285	0.0440
NRC-Canada #2	3	24.2	297	0.0452
NRC-Canada #2	4	36.0	309	0.0449
NRC-Canada #2	5	48.9	322	0.0485
NRC-Canada #2	6	59.8	333	0.0500

Analysis

The analysis of the thermal conductivity data in Tables 3 and 4 was approached from a statistical and engineering perspective. Initially, the thermal conductivity data were fit to the following model

$$\lambda = \beta_0 + \beta_1(T-297) + \beta_2 z + \beta_3(T-297)z + \epsilon \tag{2}$$

where T is the mean specimen temperature in Kelvin [K], β_i are regression coefficients, and ϵ is a

random error term. An indicator variable, z, has been introduced; where $z = 0$ identifies the data from NIST, and $z = 1$ identifies the data from NRC-Canada. (The selection of these values for z was purely arbitrary.) Therefore, when $z = 0$ the model becomes

$$\lambda = \beta_0 + \beta_1(T-297) + \epsilon$$

and when $z = 1$ the model becomes

$$\lambda = (\beta_0 + \beta_2) + (\beta_1 + \beta_3)(T-297) + \epsilon$$

At 297 K, the intercepts (i.e., offset) of the lines for the two data sets are equal when $\beta_2 = 0$ and the slopes of the two data sets are equal when $\beta_3 = 0$. To test these hypotheses, 95 percent confidence intervals (CI) were constructed for β_2 and β_3. The form of the confidence interval is

$$\hat{\beta} \pm t_{0.975,n-p}\, sd(\hat{\beta}_i) \qquad (3)$$

where n is the number of data points, p is the number of parameters in eq (2), $t_{0.975,n-p}$ is 97.5 percentile of the Student's t-distribution with $n - p$ degrees of freedom, and $sd(\hat{\beta}_i)$ is the standard deviation of the estimated regression coefficient $\hat{\beta}_i$. If the interval defined by eq (3) contains zero, then the coefficient of interest is statistically insignificant at $\alpha = 0.05$, where 95 percent = $100(1 - \alpha)$ percent. Here, α, the significance level of the test, is the probability of concluding that the estimates for the regression coefficients (i.e., offsets and slopes) differ, when in fact they do not. Estimates for $\hat{\beta}_i$ were determined by regression analysis. From Table 5, at $\alpha = 0.05$, we conclude that there is a statistically significant difference (i.e., nonzero) between the offset estimates for the thermal conductivity data for the glass-fiber board. None of the other coefficients for β_2 and β_3 are statistically significant.

Table 5 -- Hypothesis Test for Statistical Significance of Regression Coefficients

Quantity	Glass-fiber Board	Fibrous Alumina Silica
$\hat{\beta}_2$	0.000472	0.000039
$\hat{sd}(\hat{\beta}_2)$	0.000053	0.000071
$t_{0.975,n-p}$	2.306	2.365
95 % CI	(+0.000351, +0.000594)	(−0.000128, +0.000207)
$\hat{\beta}_3$	−0.0000048	−0.0000078
$\hat{sd}(\hat{\beta}_3)$	0.0000050	0.0000041
$t_{0.975,n-p}$	2.306	2.365
95 % CI	(−0.0000165, +0.0000068)	(−0.0000174, +0.0000018)

In the analysis, an engineering assessment of the uncertainties due to the measurement process was also considered. As mentioned above, the relative (expanded, $k = 2$) uncertainty estimates for the measurements by both laboratories were approximately ± 1.0 %. Further, the stated imprecision for standard test methods is ± 2 % to ± 5 % [1,2] at ambient conditions for homogeneous materials. Given that the difference in the offset estimates for the two data sets for glass-fiber board was less than currently accepted uncertainty estimates, the laboratory data for each material was analyzed collectively.

The data from Table 3 and Table 4 were subsequently fit to eq (2) with $\beta_2 = \beta_3 = 0$. The final equations for glass-fiber board and fibrous alumina silica are shown in eq's (4) and (5), respectively and are also plotted in Figure 1.

$$\hat{\lambda} = 0.03364 + 1.080\times10^{-4}(T-297) \quad \textbf{(4)}$$

$$\hat{\lambda} = 0.04539 + 1.264\times10^{-4}(T-297) \quad \textbf{(5)}$$

The residual standard deviations for the above fits for glass-fiber board and fibrous alumina silica were 0.00024 $W{\cdot}m^{-1}{\cdot}K^{-1}$ and 0.00013 $W{\cdot}m^{-1}{\cdot}K^{-1}$, respectively, which are quite small. The relative standard deviations multiplied by 2 for the fitted models of glass-fiber board and fibrous alumina silica were 1.4 % and 0.5 %, respectively. The adequacy of the fits was further examined by plotting the individual deviations (δ) from the model as defined by

$$\delta = \lambda - \hat{\lambda}. \quad \textbf{(6)}$$

Individual deviations versus T for glass-fiber board and fibrous alumina silica are shown in Figure 2. Figure 2a graphically illustrates the nonzero offset that was observed in Table 5 for the two laboratories. The deviations in Figure 2b do not indicate any trends, signifying a satisfactory fit.

Conclusions

A round-robin interlaboratory comparison has been conducted between guarded hot plate apparatus at NIST and NRC-Canada for two thermal insulation materials, glass-fiber board and fibrous alumina silica. A single pair of specimens of each material was circulated to both laboratories. The nominal bulk density and thickness for the specimens of glass-fiber board and fibrous alumina silica were 160 $kg{\cdot}m^{-3}$ and 25.1 mm, and 290 $kg{\cdot}m^{-3}$ and 25.2 mm, respectively. Measurements of thermal conductivity were conducted over a temperature range of 273 K to 340 K. Regression analyses of the data revealed a statistically significant (at $\alpha = 0.05$) difference, on the order of 1 %, for the offset estimate between the laboratory data for glass-fiber board at 297 K (24 °C). The reason for the offset is unknown. There was no offset noted for the data for fibrous alumina silica. It should be noted that the offset difference noted in this study is within the stated experimental (expanded, $k = 2$)

uncertainties for the respective apparatus, nominally ± 1 %. Moreover, the offset difference was well within the precision statements of ± 2 % to ± 5 % provided in standard test methods for guarded hot plate apparatus [1,2]. In conclusion, it should be stated that although the agreement between NIST and NRC-Canada can be considered quite satisfactory, continued improvement in measurement technology is always desired and therefore continued interlaboratory investigations are suggested.

References

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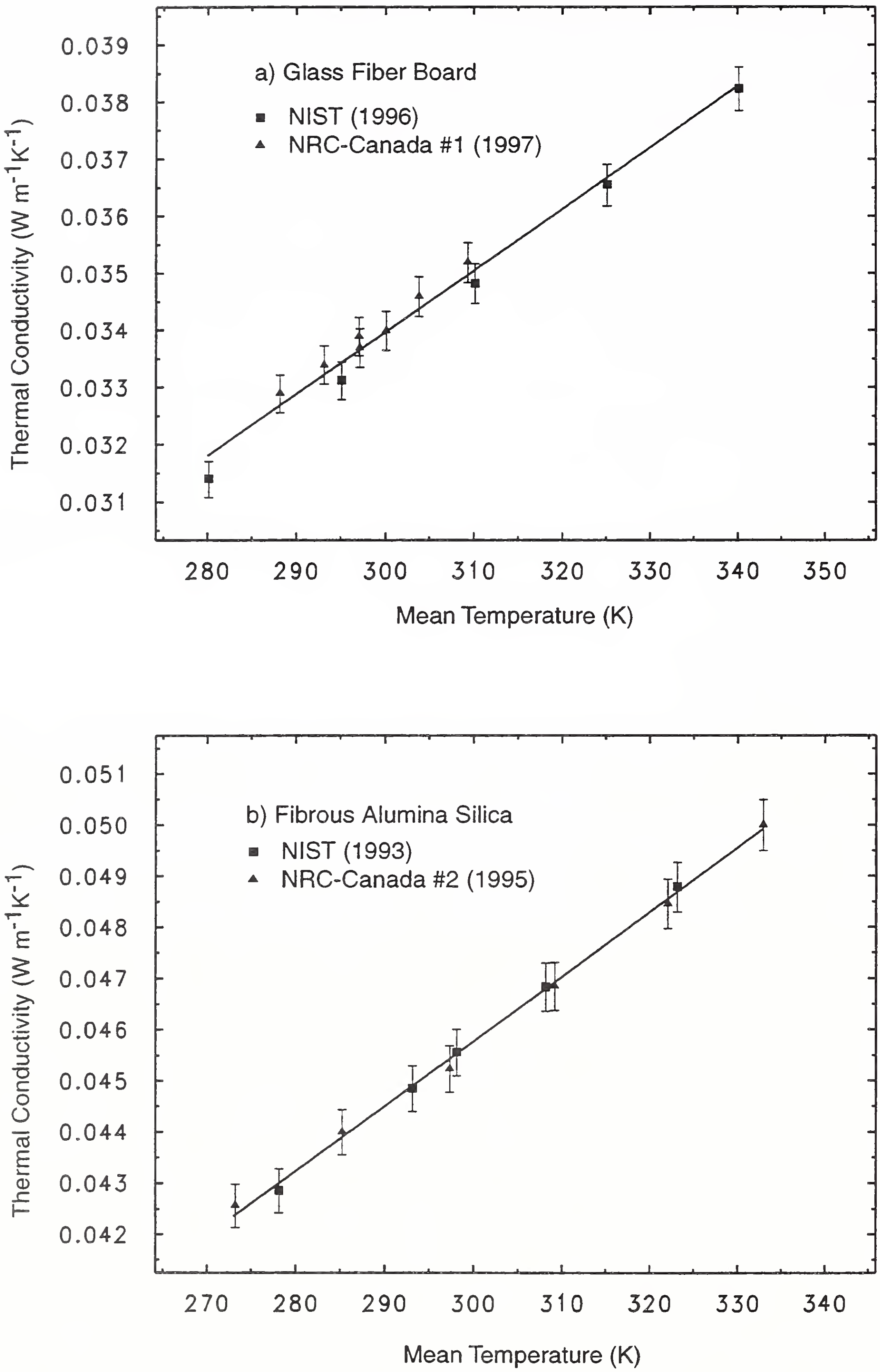

Fig. 1 - Thermal conductivity as a function of mean temperature; the solid line represents the regression analysis for all data; relative expanded uncertainty bars of ± 1 percent are included for each data point

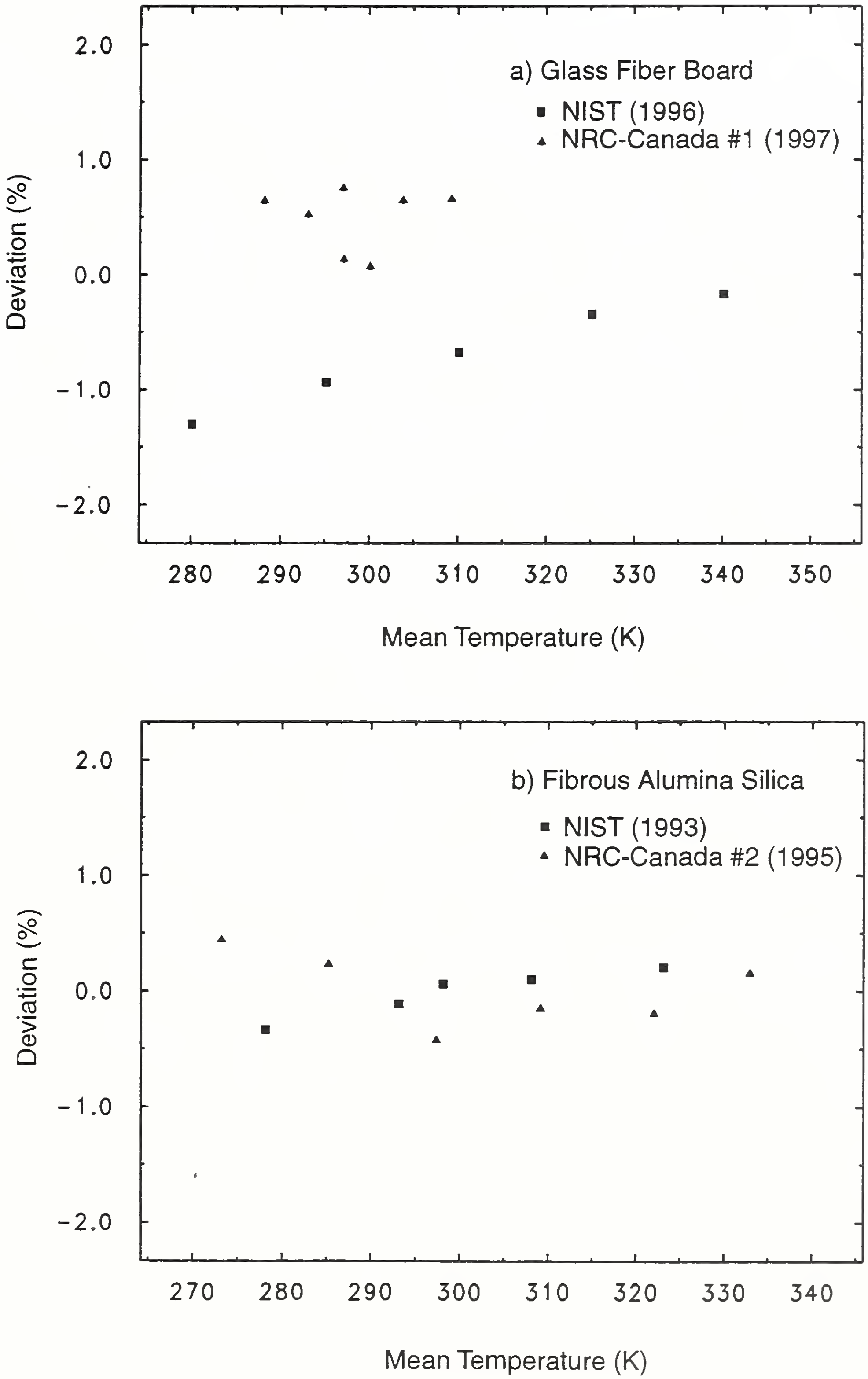

Fig. 2 - Deviations versus mean temperature